CHAPTER 3

SAMPLE PREPARATION

3.1 BASIS OF DETERMINATION

Determine sunflower seed NMR oil on a representative portion cut from the sample after the removal of foreign material. Report the sunflower seed NMR oil percent on a 10 percent moisture basis (mb).

3.2 CLEANING SAMPLES

Use a Boerner divider to obtain a representative sample portion of approximately 50 to 55 grams. Mechanically clean the sample portion using a Carter Dockage Tester operated as specified in the Grain Inspection Handbook, Book II. Sunflower seeds and dehulled seeds that pass over the riddle and the material that passes over the No. 3 and over the No. 8 sieves are combined to form the mechanically cleaned sample. Handpick the mechanically cleaned sample portion to remove all matter other than sunflower seed and dehulled seeds.

3.3 PORTION SIZE

The sample used to determine the NMR oil must not extend above the top of the Radio Frequency (R.F.) coil. For a 150 ml NMR sample tube, a depth of 50.8 to 63.5 mm (2 to 2**2** inches) must be observed.

Before drying the handpicked sample portion, check the sample volume using a marked NMR sample tube. If the sample volume does not fall within the required range, adjust the sample size until it does. If the deviation is large, use a Boerner divider to adjust the sample size.

3.4 DRYING SAMPLES

The technique used to determine sunflower seed oil is based on measuring the number of electromagnetically activated hydrogen atoms in a sample. Therefore, liquid hydrogen atom sources other than oil, such as moisture, must be removed prior to NMR oil determination.

Perform the following procedures for drying the sunflower seed sample to remove moisture:

a. Preheat the moisture oven to 130EC and check the oven temperature with a calibrated thermometer. The oven temperature must be 130EC $\pm$ 2EC before drying samples.

b. Place the handpicked sunflower seed sample in a moisture dish and record the dish ID number.

c. Place the sample in the preheated moisture oven with the lid under the dish. Dry the sample for 3 hours. Begin timing the drying once the temperature reaches 130EC $\pm$ 2EC.

3.5 COOLING SAMPLES

After drying, the sunflower seed samples must be stabilized to room temperature before performing NMR oil determination. In addition, room and sample temperature must be within $\pm$ 0.5EC from the temperature recorded during restandardization.

Allow samples to cool to room temperature using one of the following procedures:

a. Desiccating Cabinet Method.

(1) Place a calibrated thermometer inside the desiccating cabinet.

(2) Immediately after removing the samples from the drying oven, place the lid on the dish and place the dish in the desiccating cabinet. Monitor the desiccating cabinet temperature.

(3) When the room and desiccating cabinet temperature are within $\pm$0.5EC, samples are ready for NMR oil determination. The cooling time will be a minimum of 3 hours.

b. NMR Sample Tube Method.

A thermometer inserted through a rubber stopper is needed to measure the temperature of at least 1 sample in a rack (maximum 10 samples per rack) as they cool. The thermometer must be inserted into the rubber stopper so that approximately 25 mm of the thermometer will be immersed in the sunflower seed sample during the cooling period.

(1) Immediately after removing each sample from the drying oven, pour the sample into a marked NMR sample tube and seal with a rubber stopper. Each rack must have at least one rubber stopper with a thermometer inserted.

(2) Place the NMR sample tube into a rack. The rack should keep sample tubes separated by approximately 20 mm.

(3) When room temperature and the thermometer reading are within $\pm$ 0.5EC, the samples are ready for NMR oil determination. The cooling time will be at least 3 hours.